UNIVERSITI PUTRA MALAYSIA

SYNTHESIS, CHARACTERIZATION AND APPLICATION OF CARBON NANOTUBES AND CARBON NANOFIBERS

MUATAZ ALI ATIEH HUSSEIN.

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By

MUATAZ ALI ATIEH HUSSIEN

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DEDICATION

بسم الله الرحمن الرحيم

اقرأ باسم ربك الذي خلق(1)

خلق الإنسان من علق(2)

اقرأ وربك الأكرم(3)

الذي علم بالقلم (4)

علم الإنسان ما لم يعلم(5)

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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May 2005

Chairman : Associate Professor Fakhru'l-Razi Ahmadun, PhD

Faculty : Engineering

Well aligned multi wall carbon nanotubes (MWCNTs), carbon nanofibers (CNFs) and other type of carbon nanostructure materials have been synthesized by a fabricated floating catalyst chemical vapor deposition (FC-CVD) method. This involved the pyrolysis of benzene-ferrocene vapor mixture. The CVD parameters (Hydrogen flow rate, reaction time and reaction temperature) were studied to selectively synthesize nanotubes and nanofibers with required dimensions. Carbon nanotubes films with a diameter of 2-50 nm and nanofiber with a diameter range from 100-300 nm were synthesized in a benzene/hydrogen atmosphere. Furthermore vapor grown carbon fibers have been synthesized with different diameters and lengths. Iron clusters that were produced from the thermal decomposition of ferrocene films were used as catalyst for the synthesis of the carbon structures.

The effects of different hydrogen flow rates (50-500 ml/min) on the morphology, quality and quantity of the product were investigated. Maximum yield and purity was obtained at 300 ml/min.

The effect of the reaction time on the purity and yield of carbon nanotubes was studied from 1 minute to 60 minutes. There was no effect of the reaction time on the average diameter while maximum yield of carbon nanotubes was achieved at 45 minutes.

The last variable was the reaction temperature, which was varied from 500 °C to 1200 °C. By controlling the growth temperature, carbon nanotubes (CNTs), carbon nanofibers (CNFs) and vapor grown carbon fiber with different structures were produced. Increasing the temperature has a remarkable effect on the size and shape of the catalyst and this in turn affected the diameter distribution and structure of the carbon materials. The carbon nanotubes were produced from 600 °C to 850 °C with maximum yield at 850 °C, while for the production of carbon nanofibers the reaction temperature was from 900 °C to 1000 °C with a maximum yield at 1000 °C. Vapor grown carbon fibers were produced at 1050 °C to 1200 °C with maximum yield at 1050 °C.

The synthesised nanotubes/nanofibers were investigated by scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

The thermal degradation kinetics of CNTs was investigated by dynamic thermogravimetry, in an air atmosphere, over the temperature range 25 - 800 °C and at constant nominal heating rate 10 °C/min. The corresponding activation energies, frequency factors and reaction orders were determined.

Homogenous distribution of MWCNTs/CNFs in natural rubber (NR) was achieved by ultrasonic assisted solution-evaporating method. Addition of 1-10 wt% of CNFs and CNTs to natural rubber as nanocomposite increased the rubber mechanical properties significantly. The properties of the composites such as tensile strength, tensile modulus, and elongation at break were studied. In addition to mechanical testing, the dispersion state of the MWNTs into NR was studied by TEM in order to understand the morphology of the resulting system. The result indicated that, by increasing the amount of CNTs and CNFs into the natural rubber the ductility decreased and the material became stronger and tougher but at the same time more brittle. The results showed that by adding 1 wt% of CNTs and CNFs to NR the stress level were increased sharply to 0.56413 and 0.54 MPa respectively compared to NR which was 0.2839 MPa. At 10 wt% the stress level of CNTs with NR were increased sharply 9 times and reached to 2.55 MPa while for CNFs it increased 4.66 times and reached to 1.33 MPa.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagi memenuhi keperluan untuk ijazah Doktor Falsafah

PENGHASILAN, PENCIRIAN DAN APLIKASI KARBON NANOTIUB DAN KARBON NANOSERAT.

Oleh

MUATAZ ALI ATIEH HUSSIEN

Mei 2005

Pengerusi : **Profesor Madya Fakhru'l-Razi Ahmadun, PhD**

Fakulti : **Kejuruteraan**

Karbon nanotiub, karbon nanoserat dan struktur karbon yang lain telah disintesis dengan menggunakan kaedah pemangkin terapung-penguraian wap kimia, floating catalyst chemical vapor deposition (FC-CVD) iaitu pernguraian benzena dan ferosin menjadi campuran wap. Parameter CVD (kadar alir hidrogen, masa dan suhu tindak balas) dikaji untuk mendapatkan kaedah penghasilan nanotiub dan nanoserat mengikut dimensi yang dikehendaki. Filem nanotiub dengan julat diameter antara 2–50 nm dan nanoserat antara 100–300 nm disentesis dalam atmosfera benzena/hidrogen. Selain itu, karbon serat turut dihasilkan dengan diemeter dan panjang yang berlainan. Kelompok ferum yang terhasil daripada penguraian haba filem ferosin digunakan sebagai pemangkin bagi menghasilkan struktur karbon.

Kesan kadar alir hidrogen yang berbeza (50-500 ml/min) terhadap morfologi, kualiti dan kuantiti produk turut dikaji. Hasil yang maksimum dan ketulenan yang tinggi diperoleh pada kadar alir hidrogen 300 ml/min.

Pengaruh bagi masa tindak balas terhadap ketulenan dan hasil karbon nanotiub dikaji antara satu minit sehingga satu jam. Masa tindak balas tidak memberi kesan terhadap purata diameter sementara hasil maksimum bagi karbon nanotiub diperoleh dengan masa tindak balas selama 45 minit.

Pemboleh ubah yang terakhir ialah suhu tindak balas yang dipelbagaikan bermula dari suhu 500 °C hingga 1200 °C. Dengan mengawal suhu tindak balas, nanotiub, nanoserat karbon serat dengan struktur yang berbeza dapat dihasilkan. Peningkatan suhu memberi kesan yang besar terhadap saiz. Bentuk pemangkin pula memberi kesan terhadap taburan diameter dan struktur bahan karbon. Karbon nanotiub dihasilkan antara suhu 600 °C – 850 °C dengan hasil maksimum pada 850 °C, manakala karbon nanoserat pada suhu antara 900 °C -1000 °C dengan hasil maksimum pada 1000 °C. Karbon serat dihasilkan antara julat suhu 1050 °C -1200 °C dengan hasil maksimum pada suhu 1050 °C.

Morfologi nanotiub/nanoserat yang telah dihasilkan kemudiannya dikaji dengan menggunakan Microskop Biasan Elektron, dan Microskop Pancaran Elektron.

Kinetik penguraian haba bagi karbon nanotiub dikaji dengan menggunakan termogravimetri dinamik. Pada keadaan udara atmosfera, dengan julat suhu antara 25-800 °C dan kadar pemanasan tetap nominal 10 °C/min, tenaga pengaktifan, faktor frekuensi dan tertib tindak balas yang berkaitan dicari.

Campuran yang sekata antara nanotiub/nanoserat dalam getah asli dapat dilakukan dengan menggunakan ultrasonik dengan menggunakan kaedah pemeluwapan larutan. Tambahan nanotiub/nanoserat sebanyak 1-10 wt % megikut peratusan berat ke dalam getah asli sebagai nanokomposit telah meninggikan sifat mekanikal getah tersebut dengan ketara. Sifat-sifat komposit seperti kekuatan regangan, modulus regangan dan pemanjangan pada titik putus dikaji. Selain itu, taburan nanotiub dalam getah asli turut dikaji dengan menggunakan Mikroskop Pancaran Elektron, Transmission Electron Microscopy (TEM) untuk lebih memahami morfologi nanokomposit tersebut.

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I certify that an Examination Committee met on 16th September 2005 to conduct the final examination of Muataz Ali Atieh Hussein on his Doctor of Philosophy thesis entitled "Synthesis, Characterization and Application of Carbon Nanotubes and Carbon Nanofibers" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

Azni Idris, PhD
Professor
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Thomas Choong Shean Yaw, PhD
Lecturer
Faculty of Engineering
Universiti Putra Malaysia
(Internal Examiner)

Robiah Yunus, PhD
Associate Professor
Faculty of Engineering
Universiti Putra Malaysia
(Internal Examiner)

Ahmad Fauzi Ismail, PhD
Professor
Faculty of Chemical and Natural Resource Engineering
Universiti Teknologi Malaysia
(External Examiner)

GULAM RUSUL RAHMAT ALI, PhD
Professor/Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 25 OCT 2005

This thesis submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee are as follows:

Fakhru'l-Razi Ahmadun, PhD
Associate Professor
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Chuah Teong Guan, PhD
Lecturer
Faculty of Engineering
Universiti Putra Malaysia
(Member)

EL-Sadiq Mahdi, PhD
Lecturer
Faculty of Engineering
Universiti Putra Malaysia
(Member)

AINI IDERIS, PhD
Professor/Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 17 NOV 2005

DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citation, which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

MUATAZ ALI ATIEH HUSSIEN

Date: 14/10/05

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